UNIVERSITI TEKNOLOGI MARA

# KINETIC BEHAVIOUR AND THERMODYNAMIC STUDY OF PEANUT OIL EXTRACTION USING ULTRASOUND TECHNOLOGY

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BACHELOR OF ENGINEERING
(HONS.) CHEMICAL

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**NUR SHAHIDAH BINTI MUSA**

Thesis submitted in fulfillment
of the requirements for the degree of
**Bachelor of Engineering (Hons.) Chemical**

**Faculty of Chemical Engineering**

**July 2019**

# AUTHOR'S DECLARATION

I declare that the work in this dissertation was carried out in accordance with the regulations of Universiti Teknologi MARA. It is original and is the results of my own work, unless otherwise indicated or acknowledged as referenced work. This thesis has not been submitted to any other academic institution or non-academic institution for any degree or qualification.

I, hereby, acknowledge that I have been supplied with the Academic Rules and Regulations for Under Post Graduate, Universiti Teknologi MARA, regulating the conduct of my study and research.

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# ABSTRACT

In the present study, ultrasound assisted extraction was utilized to extract oil from peanut. There are four type of main operating parameter such as type of solvents, extraction time, extraction temperature and solvent to solid ratio were investigated. The optimum conditions were found at 15 min reaction time, extraction temperature of 40°C, solvent to solid ratio of 20:1 and ethyl acetate as solvent. While, the maximum oil yield is 43.8%. Evaluation of kinetics of oil extraction process was done by using hyperbolic model. High values of coefficient of determination ($R^2 \geq 0.9515$) and low values of the mean relative percentage deviation (MRPD $\leq$ 0.2759) were obtained, showing the model used were suitable to describe the kinetic of peanut oil extraction. Furthermore, the influence of two parameter of extraction which are extraction temperature and solvent to solid ratio on oil yield were studied. It was found that the oil yield increased with increasing of extraction temperature and solvent to solid ratio. The activation energy (Ea) was calculated as 13.402 kJ $mol^{-1}$ and the extraction of peanut oil was an endothermic process.

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Finally, this thesis is dedicated to my both parent for the vision and determination to educate me. This piece of victory is dedicated to both of you.

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# LIST OF SYMBOLS

**Symbols**

| | |
|---|---|
| °C | Celcius |
| eV | Electronvolt |
| K | Kelvin |
| A | Independent factor |
| T | Temperature |
| wb | Weber |
| W | Watt |
| % | Percentage |

# LIST OF ABBREVIATIONS

**Abbreviations**

| | |
|---|---|
| MAE | Microwave assisted extraction |
| UAE | Ultrasound assisted extraction |
| SC-$CO_2$ | Supercritical Carbon Dioxide |
| OFAT | One factor at a time |
| GC-MS | Gas chromatography–mass spectrometry |
| UV | Ultra Violet |
| MRPD | Mean relative percentage deviation |
| EI | Ionization voltage |
| RI | Retention indices |
| US | Ultrasound |

# LIST OF NOMENCLATURE

**Nomenclature**

| | |
|---|---|
| $C_S$ | Extraction capacity |
| Ct | Experimental oil yield |
| $K_w$ | Mass transfer coefficient at washing |
| $K_d$ | Mass transfer at diffusion |
| $C_o$ | Initial concentration |
| $C_{eq}$ | Concentration Equilibrium |
| $K_2$ | Peleg's capacity constant |
| $C_1$ | Extraction rate |
| $C_2$ | Maximum extraction yield rate |
| q | Experimental oil yield |
| A | Independent factor |

# CHAPTER ONE:

# INTRODUCTION

## 1.1 Research Background

Vegetable oil is oil that is extracted from more than a few kinds of fruits, seeds, grains, and nuts. The most popular oils are made from canola, coconut, corn, cottonseed, olive, palm, palm-kernel, peanut, safflower, soybean, and sunflower. People in many areas started to process vegetable oils thousands of years ago, making use of whatever meals stuffs they had on hand to gain oils for a variety of cooking purposes. Early peoples realized to use the sun, a fire, or an oven to heat oily plant products until the flowers exuded oil that may want to then be collected. The Chinese and Japanese produced soy oil as early as 2000 before century, whilst southern Europeans had begun to produce olive oil by 3000 before century. In Mexico and North America, peanuts and sunflower seeds have been roasted and crushed into a paste before being boiled in water and the oil surface was then skimmed off. Africans additionally grated and beat palm kernels and coconut meat and then boiled the resulting pulp, skimming the warm oil off the water. Some oils have become accessible only recently, as extraction technology has improved. Corn oil first became available in the 1960s (Perrault, 2017).

Peanut oil used to be first popularised in the 18th century as a result of the research and promoting of American inventor and agricultural professional which is George W Carver (Stalker, 1984). Carver realised that peanut plants should be planted in rotation with the popular southern crop, cotton, to substitute the nitrate depleted from the soil. According (Perrault, 2017), oil content material is frequently considered to be about 48-50% oil and typically independent of market type of increase habit. Triacylglycerol content of the oil is usually 95%. The maturation of the seed effects in make bigger in total oil, triacylglycerol and ratio of oleic acid to linoleic acid (O/L). Whilst free fatty acids, polar lipids, monoacylglycerols, and diacylglycerols decreases. groundnut oil or acknowledged as *Arachis Hypogaea L* carries an excessive proportion of unsaturated fatty acids, in specific oleic (18:1) and linoleic (18:2), palmitic (16:0), stearic(18:0), arachidic (20:0), 11-eicosenoic (20:1), behenic (22:0) and lignoceric

(24:0) are also found in peanut oil , however solely palmitic acid exceeds 10% (Perrault, 2017).

The traditional solvent extraction which has been proposed for decades, requires extended extraction times and tremendously massive quantities of solvent. Many methods which includes conventional method such as steam distillation, hydrodistillation and solvent extraction, and as properly as emerging revolutionary and promising techniques such as supercritical fluid extraction, microwave assisted extraction (MAE) and ultrasonic assisted extraction (UAE) are utilized to the extraction of essential oils (Ying Wang et al., 2018). Conventional vegetable oil extraction is carried out with the aid of pressing or solvent extraction. Solvent oil extraction is the most environment friendly method, however, its application provides some industrial negative aspects such as plant safety problems, emissions of volatile organic compounds into atmosphere, excessive operation expenses and bad quality products caused by using high processing temperatures (Buenrostro & LópezMunguía, 1986; del Valle & Aguilera, 1999). However, ultrasound-assisted extraction (UAE) and microwave assisted extraction (MAE) are now identified as environment friendly extraction techniques that dramatically reduce down working times, growing yields and the high-quality of the extract. Although notably exploited on the laboratory scale, both have already found industrial purposes in this field, particularly the latter (Cravotto et al., 2008).

The mechanical results of ultrasound provide a higher solvent penetration into cell materials therefore improving mass transfer, whilst the disruption of biological cell walls enables the release of its content material (Bonfigli, Godoy, Reinheimer, & Scenna, 2017).Another advantage of ultrasonic extraction is performing extraction at lower operation temperatures, which preserves greater bioactive compounds of the extracts ultrasound (S. Haji Heidari, 2017), decrease reaction time and usage of amount of solvent (Bonfigli et al., 2017). For supercritical fluid method, it is used to extract excessive added value compounds from many distinctive sources. Supercritical $CO_2$ (SC-$CO_2$) is one of the most common supercritical solvents due to its properties: moderate critical point (critical temperature 31.7 °C and critical pressure about 7 MPa), non-toxicity, non-explosivity, low fee and high selectivity to non-polar molecules such as oils. Its solvent properties can be modified dramatically with small adjustments in pressure and temperature (M. Mukhopadhyay, 2000). SC-$CO_2$ additionally separates

without difficulty from the extract, as soon as the pressure is released, leaving no traces in the extract. All these properties make SC-$CO_2$ a potentially profitable solvent for the extraction of oil from quinoa (M.G. Bernardo-Gil et al, 2011).

Throughout the world, frying, and cooking represent by the greatest usage of peanut oil. Peanut oil is additionally used in preparation of shortenings, margarine and mayonnaise. Some salad oil use is discovered and the peanut oil is suitable for use in dressings due to the fact of the length of time solids are held in suspension in the oil. Peanut oil solidifies from 0-3$^{o}$C and for this reason does no longer meet the strict terminology for salad oil. A high smoke point of 229.4 $^{o}$C is one the principal motive peanut oil used in deep-fat drying. The excessive temperature lets in food to cook rapidly with a crisp coating and little oil absorption. In India, peanut oil is used in the manufacture of Vanaspati, a vegetable ghee substitute. Peanut oil is the predominant ingredient in this hydrogenated fats that resembles natural butter and ghee in look and texture. Speciality makes use of peanut oil consist of use in cleaning soap to supply long lasting lather, however due to the fact the oil is especially unsaturated it is prone to rancidity. Groundnut oil is additionally considered as awesome emollient or non-drying, skin conditioning oil and other, that comparable in these properties to olive and castor oil. Distinctly aromatic peanut oil and peanut extract are high-value products with a strong roasted peanut taste and nut aroma. These products have applications in taste compounds, confection, sauces, baked goods, and flavour compound bases.

Also, ground nut oil is packed with Omega 3 fatty acids and protein which are responsible for keeping the skin young. Omega 3 fatty acids reduce inflammation in the body to prevent skin eruptions. Peanut oil lowers the risk of developing skin cancer. It also give hydration to the skin to treat dry and scaly skin. Eczema, rashes, painful inflammation and wrinkles are some skin disorders that can be treated with the use of peanut oil. Some benefits that users of ground nut or pea nut oil gain are; it guards the skin from the harsh Ultra Violet (UV) rays of the sun to protect against sun burn and skin damage. Ground nut oil helps fight against skin disorders such as rashes, acne, eczema, et cetera. . It also leads to speedy healing of wounds and bruises on the skin. Ground nut oil massage can energize your body and help to get rid of joint problems. Prolonged use of ground nut oil on skin yields good results.

## 1.2 Problem Statement

Peanut oil is the one of example of vegetable oil. It is also the healthiest oil that mostly used in China, USA, and Australia for cooking. It simply used because it does not absorb the flavours from the foods that are fried in it, meaning that the multiple food types can be fried in the same batch of oil without cross contamination of flavours. Additionally, peanut oil is naturally trans-fat-free, cholesterol free, and low in saturated fats. Besides that, peanut oil is also used in medical industry to produce vaccines. Due to the multipurpose of peanut oil, the rate of demand of peanut oil had been increase rapidly. According to National Edible Oil Distributor Association, the amount of peanut oil production is over 22 million for year of 2017 to fulfil the massive demand of consumers. The rate production will be increase with time to time, so research has been conducted to overcome and fulfil the massive demand of consumers. Alternatively, an ultrasonic method is used to remove oil as it has its own advantages. To scale up the process design and analysis process can be done by using mathematical model. The implementation of mathematical model can optimize, control and provide the useful information data to scale-up the equipment. The mathematical tool that reflects physical behaviour of the actual structure and experimentation observation. Hence it can be used as a simulation tool and then in the industrial application of this process (Toda, Sawada, & Rodrigues, 2016).

## 1.3 Research Objectives

The objectives of this study are:

1. To optimize extract parameter of peanut oil using one factor at a time method (OFAT).
2. To evaluate the kinetic of ultrasound assisted extraction (UAE) of peanut oil using mathematical model.
3. To investigate thermodynamic of the extraction oil process.
4. To analyse chemical constituent of extracted oil.

## 1.4 Scope Study

The research scope of production of the peanut oil by using solid liquid extraction that assisted by ultrasonic. Peanut will be ground to measurement of 0.5-0.75mm. The powder of peanut will be mixed into solvent in the conical flask. The conical flask and its content will be immersed into the ultrasonic cleaner bath. After the extraction, the conical flask that is containing the resulting mixture will be centrifuged to remove solid and evaporated by using rotary evaporator to remove solvent. OFAT will be done to determine the optimum parameter for oil extraction. The fatty acid content in the oil will be determined by using GC-MS analysis. The sample will be analysed by using mathematical model and thermodynamic equation.

## 1.5 Thesis Organisation

This thesis is divided into five (5) chapters which are included as follows:

1. Chapter 1: describes about general background of vegetable oil, problem statement of this study, the objectives of the research, scope and the organisation of the study.
2. Chapter 2: deals with review of literature. It explain about peanut oil with aspect of botanical, geographic and potential application. In this chapter also explain about method of extraction, optimisation method, extraction kinetic and summary of this part.
3. Chapter 3: provides the research methodology adopted for this study to be carried out which are extraction method and analysis of the sample.
4. Chapter 4: present the result and data. Then, discuss the result that consist of one factor at a time (OFAT), content of the peanut oil, kinetic extraction and thermodynamic analysis.
5. Chapter 5: conclude all the chapters.

# CHAPTER 2:

# LITERATURE REVIEW

## 2.1 Peanut

Peanut is known as groundnut, its scientific name can be classified as *Arachis hypogaea* and it also known as earth nut because of the seeds develop underground. The groundnut belongs to the pea and bean family and is a legume. Although it is regarded as nut due to the fact of its excessive nutritional value. Species in the genus Arachis are amazing from most different plants by using flowering above ground, however producing fruits under the soil surface. Peanut is a member of the Fabaceae, tribe Aeschynomeneae, subtribe Stylosanthinae in the genus Arachis. Only A. hypogaea has been domesticated, though quite a few species have been cultivated for their seed (A.villosulicarpa Hoehne and A. stenosperma Krapov. and W. C. Gregory) or forage (A. pintoi Krapov. and W. C. Gregory and A. glabrata Benth). The domesticated species used to be described by Linneaus in 1753 as Arachis (from the Greek "arachos," which means weed) and hypogaea (meaning underground chamber). The cultivated groundnut (Arachis hypogaea L.) is an historical crop of the New World, which originated in South America (southern Bolivia/north west Argentina region) the place it was once cultivated as early as a thousand B.C.(Cueto, Carrasco, & García, 2008)

Crop distribution to Africa, Asia, Europe and the Pacific Islands passed off possibly in the sixteenth and seventeenth centuries with the discovery voyages of the Spanish, Portuguese, British and Dutch (Krapovickas, 1973; Gregory et al., 1980; Hammons, 1982; Isleib et al., 1994). There are two-seeded types originating from Brazil had been taken to Africa whereas three-seeded types originated from Peru and have been transported from the west coast of South America to China and islands in the western Pacific (Hammons, 1982). Peanut is cultivated around the world in tropical, sub-subtropical and warm temperate climates. About 13.5 million hectare are grown in Asia, 5.3 million hectare in Africa, 1.2 million hectare in the Americas, and 0.1 million ha in other components of the world (Carley and Fletcher, 1995). India and China are the greatest producers of the crop. Average yields on a world scale improved barely from 0.93 MT/ha in the 1970S, to 1.08 MT/ha in the 1980s, and 1.15 MT/ha in the

1990s (Carley and Fletcher, 1995). The global production is 29 million tonnes of pods. India, China, and the United States are the leading producers and grow about 70% of the world's groundnuts.

Six gene centres have been recognized in South America, with Bolivia (southwest Amazon) as the important centre, and the 5 secondary centres corresponding to the Guarani location (Paraguay-Parana), Goias and MinaGerais place of Brazil (Tocantins, Sao Francisco), Rondona and northwest Mato Grosso in Brazil (south Amazon), Peru (upper Amazon and west coast), and northeast Brazil areas (Krapovickas, 1973; Gregory and Gregory, 1976). Arachis hypogaea is an allotetraploid (2 n = four x =40) species which probable advanced from two diploids in part Arachis. Although a quantity of species have been proposed as progenitors, A. duranensis Krapov. and W.C. Gregory and A. ipaensis Krapov. and W.C. Gregory show up to be the most probable ancestors (Kochert et al., 1996). Cytogenetic proof for two genomes in A. hypogaea got here first from Husted (1936) who discovered one considerably smaller chromosome pair, and later from (Stalker and Dalmacio, 1986) who karyotyped accessions in each subspecies (Stalker, 1984).

Qualitatively inherited characteristics in A. hypogaea also show up to be managed via duplicate loci, which helps the speculation that this is an allotetraploid species (Wynne and Coffelt, 1982). According to Garcia et al. (1995) illustrated that the two genomes of A. hypogaea can be separated the usage of RFLPs. Thus, A. hypogaea is a segmental allopolyploid species and probable originated from two intently associated species with similar genomes. This conclusion used to be supported through Garcia et al. (1995) who reported that genes from a single diploid species can be introgressed into each genomes of A. hypogaea. Genetic characteristics in A. hypogaea have been reviewed by Hammons (1973) and Wynne and Coffelt (1982), however very few linkage organizations have been reported. Wynne and Coffelt (1982) concluded that most agronomically important characteristics in peanut are quantitatively inherited. Additive genetic variance is the principal issue of genetic variance for economically-necessary traits. Thus, early era trying out correctly put off many undesirable crosses, but unfortunately, yield has a low heritability and extensive genotype X environmental interactions exist.

### 2.1.1 Botanical Description

Ground nut plants growing about 0.5 m of tall or length and will be more during growing season. Peanut plant need about at least five months to grow up in warm weather with rainfalls. Flower of peanut plant are always in colour yellow with reddish veins and tubular calyx, clustered in groups of up to 6 flowers and sometimes the colour of flower will be white in certain cases. After pollination, the flower stalks elongate and push the developing pods into the ground, so that the fruit must be dug up from the soil to be harvested. Flowers consist of ten androecium, with 5 anthers being elongated and the last five (5) being extra globular and small. The peanuts are pulled from the floor with the aid of exclusive machinery and turned over to dry in the fields for several days. The leaves are alternate and compound, with 4 ovate to oblong leaflets, up to 6 cm long (2.25 in) (Daoudi, 2000). Mostly, peanut contains more than 50% of oil. The fruit is an indehiscent legume (a pod that does no longer have sutures or split open freely), commonly containing 1 to 3 gentle seeds, sometimes it can be 6 of seeds, each included with a reddish brown, papery membrane. The mass of a typical seed is around 1.3 g and sometimes the small seed is about 0.047 g (Stalker, 1984).

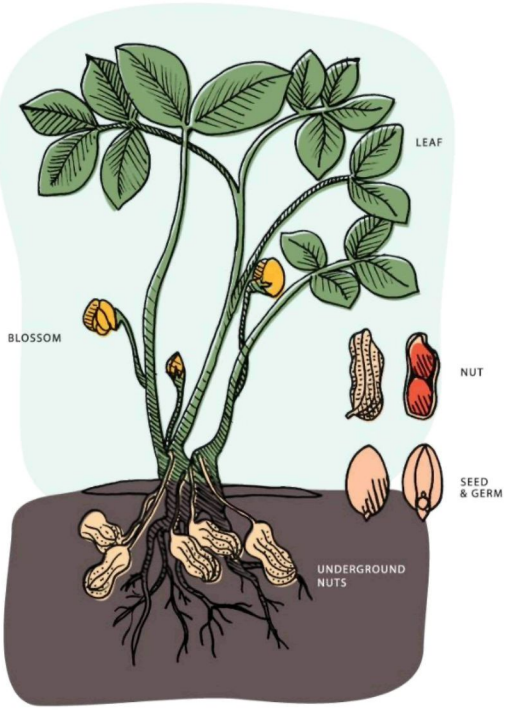


*Figure 3-1* Visualization of Peanut Plant

### 2.1.2 Geographic Distribution

The first-rate soil suited to peanut production is well-drained, light coloured, loose, friable, sandy loam that carries excessive levels of calcium, a reasonable quantity of natural matter, and with reasonable to slightly acidic pH ranging from 5.8 to 6.5. Optimum peanut production can be done in areas with a depth of 4 to 60 centimetres of soil which is friable with sandy loam or clay loam subsoil. The area of cultivation should be plow and harrow so that the seeds can easily germinate, and acquire appropriate soil tilt. Set furrows 50-60 centimetre apart to allow relative ease of weeding, cultivation and spraying besides demanding the developing crop (Stalker, 1984).

### 2.1.3 Potential application

The usage of nuts is diverse and all parts of the plant can be used. According to Star Online ("Peanut roots and coconut water - Health | The Star Online," n.d.), The seed coat of peanut, in particular those red in colour, is used to deal with issues regarding blood and bleeding. Peanut shells are included in prescriptions for treating cough. Boiling clean peanut leaves in water and consuming the concoction earlier than bedtime is to make certain a right night's sleep. Additionally, young leaves and tips can be cooked as green vegetable. According to (Martin, F. W. ; Ruberte, 1975), the young pod of peanut can be as vegetable and cooked with other vegetables. The vines with leaves are a great excessive protein hay for horses and ruminant livestock. The pods or shells serve as excessive fibre roughage in livestock feed, fuels, mulch, and are used in manufacturing particle board or fertilizer. The most important part of peanut is its seed, mostly in American, they consume peanut as roasted seeds or peanut butter compared to use as oil elsewhere in the world. About 4 million pounds of peanuts per day were consumed by Americans. This is because the seed contains about `36 to 54% oil and 25 to 32% protein (Knauft & Ozias-Akins, 1995). Most commercially grown peanuts are used for the extraction of their oil which is used in cooking. The side product of oil extraction is a pressed cake which is used as an animal feed and additionally in the manufacturing of peanut flour. Peanut is also used for aphrodisiac purposes, inflammation, cholecystosis, nephritis and decoagulant.

## 2.2 Extraction Method

### 2.2.1 Solvent oil extraction

Solvent extraction is an extra efficient technique of extracting the oil from the seeds which takes place both in batch or continuous process. The rate of extraction depends on the thickness and surface area of the seed flakes, the working temperature, the type of solvent and the moisture content of the seeds (Ward JT, Basford WD, Hawkins JH, 1985). Solvent extraction has been reported to produce high yields oil but uses a lot of energy due to the long extraction time compared to the method of mechanical extraction. With solvent extraction of about 99.3% of the oil content in the seed was extracted compared with 75-85% with mechanical press (Yan, Fang, Long, Zhu, & Juan, 2005). Subsequently, this technique is often used in agricultural chemistry to extract oil, earlier than turning into an essential apparatus for solid liquid extraction in different fields like (Lakshmana Rao, Gupta, Bhaskar, & Jayaraj, 2002), and foodstuffs (Schmarr, Koschinski, Sang, & Slabizki, 2012). Nowadays solvent extraction is mainly used as a reference technique in many laboratories for extraction of oil from oilseed and a range of materials. Currently, most of the oilseed industries used hexane as a solvent, however more than a few solvents are presently also been viewed (Materials, 1984).

(Ofori-Boateng, Keat Teong, & Jitkang, 2012) had studied on exergy analyses of Jatropha curcas oil extraction methods. The resulting from the experiment are 94.7% of the total input exergy for solvent extraction are the products' exergy. Afterwards, solvent extraction of J. circa oil is 79% efficient based on thermodynamics. Based on (Ixtaina et al., 2011), the experiment is carried out to investigate the oil yield, fatty acid composition and the physicochemical and quality characteristics of chia seed oil. From result of the oil extraction, the oil yield is about 33.6% , while the content of oil are 6.2% of Palmitic acid (16:0), 3.0% of Stearic acid (18:0), 5% of Oleic acid (18:1), 0.5% of Vaccenic acid (18:1), 19.7 of Linoleic acid (18:2), and 65.6% of α-Linolenic (18:3). Then, refractive index is ranged from 1.4763 to 1.4798. The colour of vegetable oils is related with the total pigment content; presence of carotenoids however no longer chlorophyll pigments was detected in chia seed oils.

### 2.2.2 Mechanical Oil Extraction

Mechanical extraction is regularly the favoured choice to extract oil from oil seeds. This method can be used for the cold pressing of oil and for seeds that include excessive quantity of oil (Oberndorfer & Lücke, 1999). There have been three eras in the extraction of vegetable oils from the variety of oil-bearing seeds, nuts and fruits, specifically hydraulic pressing, continuous screw pressing and solvent extraction (Bredeson, 1978). Mechanical pressing is viewed as the preferred technique to extract oil from seed specifically in the rural areas of a growing areas (Achten et al., 2007). Mechanical extraction is the oldest and generally used extraction method which can be operated on both batch and continuous processes. The normally used technique is the batch which is very slow and inefficient in the use of labour and rate of oil yield. Screw presses or expellers are at present designed for continuous extraction process (Evon P, Vandenbossche V, Pontalier PY, 2009). The different strategies provide too much complexity for application in rural areas. Maximum oil recovery was reported to be 79% for BT50 screw press and 87% for Sayari strainer press after twin passing. Concerning the optimization of oil extraction efficiency, neither the impact of distinct settings of the screw press nor the resulting based elements were reported in different studies (Karaj & Müller, 2011).

(Karaj & Müller, 2011) carried a research on extraction of Jatropha curcas L. seeds. The experiments were carried out with mechanical screw press Komet D85-1G by manipulated different screw presses such as R8 or R11. The highest oil recovery of 89.4% (m/m) was achieved by using R8. Oil recovery decreased and oil content in press cake and seed material throughput was increased when rotational speed of screw press was greater. (Subroto, Manurung, Heeres, & Broekhuis, 2015) investigated on extraction of oil from Jatropha curcas L. kernel. Experiment is done to study the influence of process parameters on oil recovery from Jatropha kernel which are compression speed, applied pressure, moisture content of sample, pressing temperature, duration of pressing, feedstock size reduction, shell removal and preheating time. By increasing moisture content of kernel sample from 2 to 6% (w.b.), it will make increasing of the Phosphorus content from 1.4 to 3 ppm and acid value of oil from 1.03 to 1.19. Increasing pressing temperatures raises the acid values and phosphorus contents of the oil. Acid value expanded with an enhancing in pressing temperature from 25 to 60°C. However a barely decreased acid value used to be found when pressing

temperature extended to 90°C. A minimal of 80% shell removal need to be accomplished to avoid the terrible impact of shell presence on oil recovery. Five minutes of preheating time used to be found to be most beneficial in growing the oil recovery.

### 2.2.3 Ultrasound Assisted Oil Extraction

Ultrasound-assisted extraction (UAE) is extensively used for extracting plant compounds in order to overcome these drawbacks (Vilkhu, Mawson, Simons, & Bates, 2008) . Increased extraction by ultrasound was associated with the propagation of ultrasound pressure waves and ensuing cavitation forces, where bubbles can explosively collapse and generate localized pressure inflicting plant tissue rupture and enhancing the release of intracellular components into the solvent (Knorr, Ade-Omowaye, & Heinz, 2002). The feasibility of using ultrasounds (US) to enhance the recovery of oil, nutritional value, physicochemical and sensorial properties have been broadly investigated and have proved to be efficient before or during the oil extraction process. Ultrasound technology also will lowering the processing cost, producing pretty pure product, eliminating some of the downstream purification steps, as nicely as the post-treatment of wastewater. (Mohamed Koubaa , Houcine Mhemdi , Francisco J. Barba & Ralf Greiner, 2016).

(Liu et al., 2017) had investigated about optimization of ultrasonic-assisted extraction of oil from the seed kernels. From the result of the experiment, the optimal extraction time was 15 min, while the higher percent of yield at condition of power at 200-400 watt and temperature from 20 to 30 °C. The unsaturated fatty acids relative to total fatty acids percentage was exceeded 85%. The main content in oil are linolenic acid was 34.12%, oleic acid was 27.87%, and total unsaturated fatty acid was more than 85%. Extraction of pomegranate oil had been carried out by (Goula, 2013). The experiment is done to decide the kinetic parameters that describe the mechanism of pomegranate seed oil ultrasound extraction and to study the consequences of a variety of parameters on extraction yield and extraction kinetics. The higher percent of yield is 59.8% with optimum temperature at 20°C, 20:1 solvent to solid ratio and level amplitude of 60%.

(Da Porto, Porretto, & Decorti, 2013) had studied about comparison between ultrasound-assisted and conventional extraction methods to extract oil from grape seed. From the experiment result, the improvement of recovery (% total oil) was of about 18% with ultrasonic bath compared to Soxhlet method. Mostly the percent composition of free fatty acid in oil with ultrasonic assisted extraction is highest than Soxhlet method. For instance, the main composition of free fatty acid in grape seed oil are Linoleic acid, Palmitic acid, Oletic acid and Stearic acid. For Linoleic acid, the composition percent was 72.36%, palmitic acid was 7.22% while Oletic acid was 16.40 and the last stearic acid was 3.17%.

### 2.2.4 Microwave Assisted Oil Extraction

Microwave is a rising technology that is used in many food processing applications to enhance their efficiency (Chandrasekaran, Ramanathan, & Basak, 2013); (Knorr et al., 2011);(Tamborrino, Romaniello, Zagaria, & Leone, 2014). For example, microwave-assisted solvent extraction (MASE) can be used in oil extraction methods as other choice to conventional solvent extraction. Rapid heating and degradation of the biological cell structure of plant tissues under microwaves provide extremely powerful extraction procedures in a shorter time (Azadmard-Damirchi, Habibi-Nodeh, Hesari, Nemati, & Achachlouei, 2010) (Clodoveo & Hachicha Hbaieb, 2013) . Microwave-assisted solvent extraction (MAE) is a modern extraction technique for essential oils, fats and oils, and has advantages over conventional solvent extraction (Cintas, Calcio-Gaudino, & Cravotto, 2012). Afterwards, MAE has shown to reduce the extraction time and minimize the environmental impact by producing lower $CO_2$ (Ferhat M., Meklati B., Smadja J., 2006); (Lucchesi M. E., Smadja J., Bradshaw S., Louw W., 2007) and requires only a fraction of the energy used in conventional extraction methods (Farhat, Ginies, Romdhane, & Chemat, 2009).

(Yanık, 2017) had carried experiment on extraction of oil from wet olive pomace by the pressurized microwave system. Olive pomace oil can be extracted in a shorter time using microwave irradiation (16 min) compared to conventional extraction techniques (120-180). The free fatty acid content of MASE oil was considerably greater compared to that of conventionally extracted oil while peroxide value was 2.6 instances lower. The outcomes of this study described that the whole phenolic content of MASE

oil was four times greater than that of commercially extracted oil. The a-tocopherol content of MASE oil was also 2.4 times greater in contrast to that of commercially extracted oil. (Pandey & Shrivastava, 2018) also done rice bran oil extraction that assisted by microwave. The value of free fatty acid in oil was found about 4.88 ± 0.26% while the value of antioxidant activity was found 1.50 ± 0.09 mg TEAA/g RBO. Major antioxidant compounds existing in rice bran oil are -tocopherol and ɣ-oryzanol.

Microwave-assisted solvent extraction of castor oil from castor seeds had been investigated by (Ibrahim & Zaini, 2018). This experiment is carried to determine the physicochemical properties and stability of oxidation of castor oil the usage of microwave-assisted solvent extraction (MAE). The maximum oil yield of 37% is obtained at 20 minutes with microwave power intensity 330 W and S / F ratio of 20:1 Afterwards, the main fatty acid composition of castor oil is ricinoleic acid and the value is about 82.62%. The second content of castor oil is linoleic acid and the percent of linoleic acid is 16.32, it is followed by palmitic acid which is 0.42%, then 0.33 % of oleic acid and lastly is 0.30 of propanoic acid. Microwave also had been use in extraction of essential oil from herbs. This study was done by (Cardoso-Ugarte, Juárez-Becerra, Sosa-Morales, & López-Malo, 2013). 0.47% is the percent of yield that obtained with the combination of 70% of power, 30 min of microwave heating and 400 mL of water. The main content of essential oil are methyl cinnamate, linalool. Eucalyptol, β-cubebene, and cadinol. For extraction of basil essential oil, the technique did not have an effect on its chemical composition, increased range of compounds have been detected in oil obtained through microwave assisted extraction.

## 2.3 Optimization Method

The one-factor-at-a-time method also acknowledged as one-variable-at-a-time technique is an approach of designing experiments involving the trying out of factors one at a time rather of multiple elements simultaneously. The benefit of this method are it requires fewer experiments, and time for the amount of information obtained and at the same time, the estimation of the effects of each factor will be more precise. Afterwards, the interaction between elements can be estimated systematically.

According to (Tian, Xu, Zheng, & Martin Lo, 2013) that had carried a experiment about extraction oil from pomegranate seed. On the experiment, the OFAT

method had been used to study optimization of ultrasound assisted extraction parameters, namely ultrasonic power at 80W – 200W, extraction temperature of 35°C-45°C, extraction time at 20 min – 40 min, and the ratio of solvent volume to seed weight (S/S ratio) which is 8mg/g to 11ml/g. From the result of experiment, when the power was elevated from 80 to 160 W, a two-fold increase, the yield of oil accelerated drastically and nearly linearly from 19.85 to 23.23%, about 3.38% increase. For the temperature, the yield of oil increased as temperature increased from 25 to 40°C, however when the temperature exceeded 40°C, the percent of oil yield become decrease. For extraction time, the percent oil yield increase rapidly for time 20min to 30min and then exceeded 30 min the percent of yield became slow and reached to equilibrium. For the solvent to solid ratio parameter, the oil yield increase from 20.74 to 21.38% with the ratio of S/S increasing from 8 to 10ml/g but the oil yield became constant when the solvent to solid ratio exceed more than 10ml/g.

Optimization method also had been used by (Sayyar, Abidin, Yunus, & Muhammad, 2009) to investigate the factors affecting oil extraction from Jatropha seeds using organic solvents. The effect of 5 major elements which are type of solvent, temperature, solvent to solid ratio, particle measurement of the meal and reaction time have been investigated to optimize the extraction operating conditions for attaining maximum oil yield. The extraction temperature used to be different from room temperature (29°C) to boiling point of the solvent whilst the reaction time was assorted between 6-9 h. The solvent to solid ratio was investigated from 4:1-7:1 and seed size was fixed at three sizes specifically below 0.5, 0.5-0.75 and above 0.75 mm. From the result of experiment, for effect of solvents which are hexane and petroleum ether, the extraction yield with n-hexane was observed to be about 1.3% extra than that of petroleum ether. While, the parameter of temperature, the percent of oil yield for both solvent increase with temperature from 25°c until boiling point of the solvent. However the oil yield decrease when exceed boiling point of solvent. For the extraction time, the oil yield become increase from time of 6h to 7h for both solvent, but the percent of oil yield become constant when exceed more than 7 h. Then, the effect of solvent to solid ratio, the percent of extracted oil has increase with solvent to solid ratio from 4:1 to 6:1 but it become slowly decrease when the amount ratio higher than 6:1. The best possible percentage of oil yield was bought from the intermediate measurement particle (0.5-0.75 mm) which is 47.3% and 46% using hexane and petroleum ether respectively.

## 2.4 Extraction Kinetic

Mathematical models were used to describe the experimental data on the extraction kinetics. Mathematical model is a very necessary tooling the delineation of the process, which approves the information of the process' variable consequences such as solid/solvent ratio, temperature and hydration level of the solvent on the rate of extraction of the compound of activity (Toda et al., 2016). There are several mathematical model that had been used in the experimental:

### 2.4.1 Second – Order Model

The second – order kinetic equation for the rate of extraction can be written as following:

$$\frac{dc_t}{dt} = k_2 (C_s - C_t)^2 \quad (2.1)$$

Where $k_2$ is the rate constant of extraction (L $g^{-1}$ $min^{-1}$) for the second order. Afterwards, grouping the equation (3.2) such as:

$$\frac{dC_t}{(C_s - C_T)^2} = k_2\, dt \quad (2.2)$$

Then, Eqn. (3.3) was integrated by using boundary conditions of $C_t = 0$ at t=0 and $C_t = C_t$ at t = t and the rearrangement as follows:

$$\frac{1}{(C_s - c_t)} - \frac{1}{C_s} = k_2 t \quad (2.3)$$

$$C_t = \frac{C_s^2 k_2 t}{1 + C_s k_2 t} \quad (2.4)$$

From the Eqn. (2,4) it can be converted to linear form suh as :

$$\frac{t}{C_t} = \frac{1}{k_2 c_s^2} + \frac{t}{c_s} \quad (2.5)$$

The extraction rate can be written as the following:

$$\frac{C_t}{t} = \frac{1}{(\frac{t}{c_s})+(\frac{1}{kC_s^2})} \tag{2.6}$$

Then, when t approaches 0, the initial rate of extraction, h can be expressed as:

$$h=kC_s^2 \tag{2.7}$$

So, by substitute the Eqn (2.7) into Eqn (2.6), the concentration of oil can be written as following:

$$C_t = \frac{t}{\frac{1}{h}+\frac{t}{C_s}} \tag{2.8}$$

Thus, the initial extraction rate, h, the second order of extraction, k, and the capacity of extraction, $C_s$ can be calculated by plotting $t/C_t$ vs t. This second – order model had been used by (Kusuma & Mahfud, 2016) to study mass transfer rate of extracted solute from the solid matrix surface to the bulk liquid phase. (Kusuma & Mahfud, 2016) had carried extraction of essential oil from sandalwood by using microwave air - hydrodistillation method. From the experiment result, $k_2$ value of the extraction of sandalwood oil is 0.2663 L $g^{-1}$ $min^{-1}$. To determine the value of $k_2$, it can be done from the graph which is intercept of the graph of $t/C_t$ against t. The value of the coefficient of determination ($R^2$) of this model is 0.9965.

(Sayyar et al., 2009) also used second – order model to study kinetic and mechanism of extraction of Jatropha Oil. Graph of $t/C_t$ against time was plotted for temperature of 341 K, 333K and 318K. From the graph, the $k_2$ value can be determined. For temperature of 318K, the value of k was 0.00042 L $g^{-1}$ $min^{-1}$, while the determination coefficient was 0.9996. For temperature of 333K, the value of k was 0.00048 L $g^{-1}$ $min^{-1}$ and the coefficient of determination was 0.9995. For temperature of 341K, the value of k was 0.0005 L $g^{-1}$ $min^{-1}$ while the coefficient of determination was 0.9997.

### 2.4.2 The So and Macdonald (1986)

The So and Macdonald (1986) model had been used in this experiment to describe the experiment data on the kinetics of extraction of peanut oil. This model is a modification from the have been proposed by Patricelli et al (1979). Two mechanism that occur in the peanut oil extraction process. At the first moment of extraction oil which is in the washing step, it can be considered that the oil in the surface of the solid fibre will be removed by using the solvent. For the second part, the extraction of the oil that remains in the cells is carried out by using diffusion method. The So and MacDonald (1986) model can be expressed as the following equation:

$$Ct = C_e^w [1- \exp ( -K_w t)] + C_e^d [ 1-\exp (-K_d t)] \quad (2.9)$$

Where, $C_e^w$ & $C_e^d$ are concentration of oil in the solvent (g of oil 100 g of solution) at equilibrium condition. While $K_w$ & $K_D$ are the mass transfer coefficient at washing and diffusion. The final concentration of in the extract at infinite time can be expressed as:

$$C_e = C_e^w + C_e^d \quad (2.10)$$

According to (Toda et al., 2016), this model had been used to study the extraction oil from the soybean. From the experiment result, extraction that used absolute ethanol at temperature of 40°C, the mass transfer coefficients which $K_w$ and $K_d$ were 0.30 and 0.02, respectively. While the value of determination coefficient was 0.9992. For temperature of 50°C, the value of $K_w$ and $K_d$ were 0.35 and 0.03, respectively. Whilst, the value of determination coefficient was 0.9995. At temperature of 60°C, the value of $K_w$ and $K_d$ were 0.29 and 0.30, respectively. While, the value of $R^2$ was 0.9996. From the discussion, higher values of the coefficient of determination that obtained, it show good agreement of the experimental data with the model proposed by So and Macdonald (1986).

### 2.4.3 First – Order Kinetic

The first-order kinetics equation can be written in differential form as follows:

$$\frac{dC_t}{dt} = k\,(C_s - C_t) \tag{2.11}$$

Where k ($min^{-1}$) is the extraction rate constant for the first order and t (min) is the extraction time. Afterwards, Eq. (2.11) integrated with the boundary condition $C_t = 0$ at t =0 and $C_t = C_t$ at t =t:

$$\ln \frac{C_s}{C_s - C_t} = k\,t \tag{2.12}$$

Eq. (2.12) was obtained can be converted into a form of linear as follows:

$$\log (C_s - Ct) = \log (Cs) - \frac{k}{2\,303}\,t \tag{2.13}$$

Then graph of log ($C_S$-$C_t$) against t will be plotted to get the slope and intercept that can be used to determine the value of the extraction rate constant for the first-order (k) and the value of the extraction capacity ($C_S$). According to (Kusuma & Mahfud, 2016), first – order model had been used to determine extraction rate of essential oil from sandalwood by using microwave air-hydrodistillation method. From the result, k value was 0.0765 and the determination coefficient ($R^2$) was 0.9158. From the discussion, the value of $R^2$ was relatively low and this kinetic model is less capable to characterize nicely the experimental results of sandalwood oil extraction with the aid of microwave air-hydrodistillation methods.

### 2.4.4 Peleg's Model

Peleg introduced the following well-known semiempirical kinetic model (Eq. 2.14) to describe the sorption isotherms of food materials (Peleg 1988):

$$C_t = C_o + \frac{t}{K_1 + K_2\,.t} \tag{2.14}$$

Because of the similarity to the shape of sorption curves, the introduced model by Peleg is widely used to explain the extraction curves of biological materials from plant sources with modifications (Planinic´ *et al.* 2005).As initial concentration of target solute was zero in the extraction solvent due to beginning with fresh solvent, $C_0$ term

was omitted from Peleg's equation. Concentration of target solute in extraction solvent was measured at corresponding time, and its behaviour was explained using the modified Peleg's equation (Eq. 2.15) shown below:

$$C_t = \frac{t}{K_1 + K_2 .t} \quad (2.15)$$

As mentioned above, the extraction occurs in two stages: first order at the very beginning and decreases to zero order in the latter phase of the process. At the very beginning of the extraction, the term, $K_2.t$ is small enough that it can be assumed zero, so the solute concentration is

$$C_t = (1/ K_1).t \quad (2.16)$$

And when $t \rightarrow \infty$, the system reaches to the equilibrium

$$C_{eq} = (1/ K_2) \quad (2.17)$$

So $K_2$ was replaced by $1/C_{eq}$ to make regression analysis, calculated its values under changing extraction conditions, where $C_t$ is the concentration of target solute at time $t$ (mg/g dw), $C_{eq}$ is the equilibrium concentration as $t \rightarrow \infty$ (mg/g dw), $K_1$ is Peleg's rate constant (min g/mg) and $K_2$ is Peleg's capacity constant (g/mg).

According to (Karacabey, Bayindirli, Artik, & Mazza, 2013) that had carried extraction of trans-resveratrol and trans-e-viniferin from grape cane. From the experiment result, for extraction of trans-resveratrol at temperature of 21 °C, 50°C, and 81.3°C, the value of determination coefficient are 0.99.while for extraction of trans-e-viniferin. At temperature of 21°C, 50°C, and 81.3°C, the value of $R^2$ were 0.99, 095 and 0.99, respectively. So, it can be concluded that this model is suitable with the extraction of trans-resveratrol and trans-e-viniferin.

*Table 3-1*
Summary of kinetic modelling

| Kinetic model | Extraction Method | Determination coefficient ($R^2$) | References. |
|---|---|---|---|
| Second – Order Model | Solvent extraction assisted by microwave | 0.9965 | (Kusuma & Mahfud, 2016), |
| The So and Macdonald (1986) | Solvent extraction | At 60°C = 0.9996 | Toda, T. A., Sawada, M. M., & Rodrigues, C. E. C. (2016). |
| First – Order Kinetic | Solvent extraction assisted by microwave | 0.9158 | (Kusuma & Mahfud, 2016), . |
| Peleg's Model | Solvent extraction. | Extraction of trans-resveratrol at 21°C - 81.6°C was 0.99. Extraction of trans-e-viniferin at 21°C = 0.99, and at 83.6°C = 0.99 | Karacabey, E., Bayindirli, L., Artik, N., & Mazza, G. (2013). |

## 2.5 Summary

Peanut oil is the one of the vegetable oil that contains excessive nutritional value. Due to that fact, the need of consumer has increase recent time. According to Interventional research constantly show that nut oil intake has a cholesterol-lowering effect, even in the context of healthy diets, and there is rising proof of advisable effects on oxidative stress, inflammation, and vascular reactivity. As fulfillment to need of consumer, a research need to be conducted to overcome this issues. Extraction assisted by ultrasonic technology is the best solution to overcome the problem. Since ultrasound has many benefits such as less consumption of solvent, reduction of extraction time and automatically it can lower cost of production. To scale for industrial, a study of extraction should be done by using mathematical model. Mathematical model

additionally can optimize, control and provide the useful information data to scale-up the equipment.

# CHAPTER THREE: RESEARCH METHODOLY

## 3.1 Chemical and Equipment

Chemicals such as acetone (Merck), ethanol (Merck), methanol (Merck) and ethyl acetate (Merck) were used as a solvent. All chemicals are of analytical grade and used without pre-treatment. The equipment used in this study are listed in Table 3.1.

*Table 3-1*
List of Equipment

| Equipment | Model/brand | Usage |
|---|---|---|
| Cutting mill | Retsch SM100 | To grind the peanut seed to small size or powder. |
| Ultrasonic cleaner | Xuba 1 | Used to assist extraction of peanut oil by varying value of frequency. |
| Centrifuge | Sigma 3-18K | To separate mixture of oil and solid residue based on density. |
| Rotary evaporator | Heidolph Laborota 400 efficient | To remove solvent that has lower boiling point than peanut oil. |
| Gas chromatography–mass spectrometry | Varian 240-MS | To determine the content of fatty acid in peanut oil. |

## 3.2 Research Methodology Flow Chart

Figure 3.1 show the outline of methodology that employed in this work.

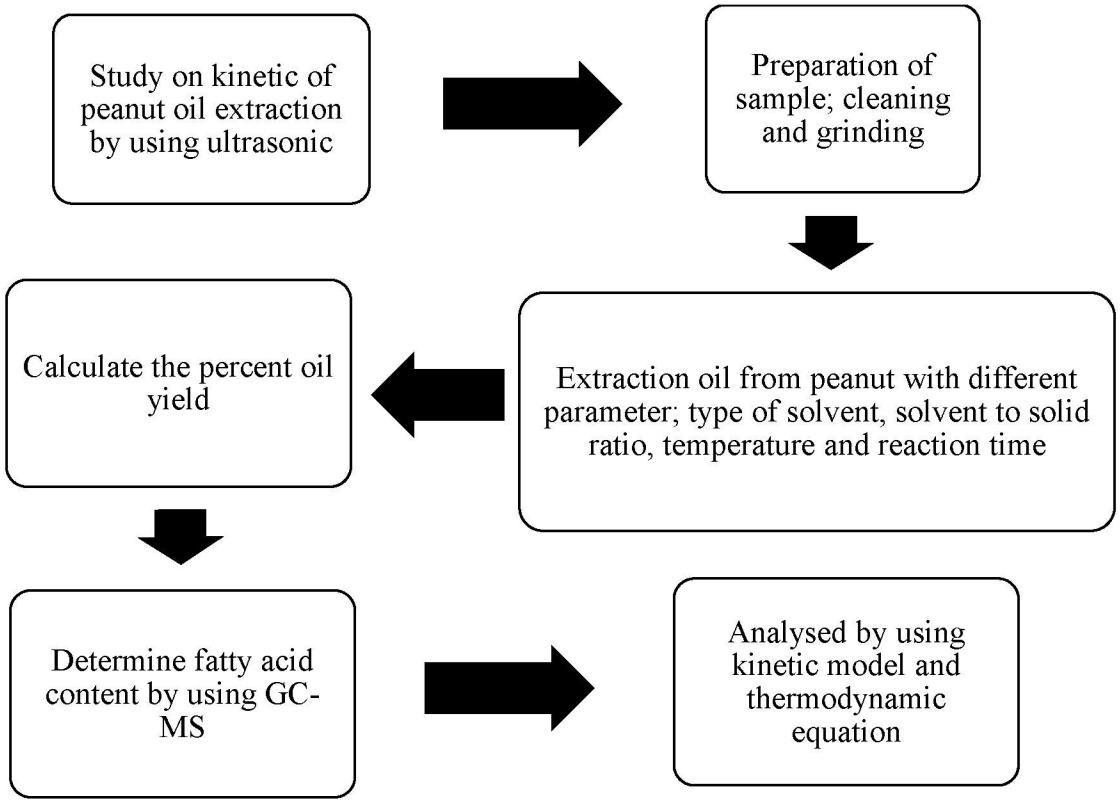


*Figure 3-1* Flowchart of methodology

## 3.3 Extraction of Peanut

### 3.3.1 Sample Collection

Peanut were purchased from nearby market of Shah Alam, Malaysia.

*Figure 3-2* Peanut seed

### 3.3.2 Sample Preparation

Prior to extraction the impurities of the seeds that consist of stones, broken, and spoiled seeds removed. Without any pre-treatment, the nut were grinded into required measurement which is 0.5-0.75mm through the usage of lab grinder. The sifted seed was stored in sealed bag for further processing.

### 3.3.3 Extraction of Oil from Peanut

5 gram of powder peanut will be mixed with 50 mL acetone in a 250mL Erlenmeyer flask and immersed into an ultrasonic cleaner bath (Xuba 1) with power of 750 Watt at temperature of 30°C. At the end of extraction, the suspension will be centrifuged for 25 min at 6000 rpm. The liquid extraction will be put into a rotary evaporator to allow solvent evaporate. The remaining oil will be weighed. Four type of solvent will be used such as acetone, ethyl acetate, methanol and ethanol. The extraction temperature was manipulated from 30°C to 70°C while the reaction time was varied between 5 min to 30 min. Then, the solvent to solid ratio was investigated from 10:1 – 50:1. These parameters were varied one at a time to determine the optimum conditions for each type of solvent.

### 3.3.4 Oil Yield

The percentage yield of the oil recovered was determined through using the use of the following equations (Zhang et al, 2008):

$$\text{Oil yield (\%)} = \frac{M_{\text{extract}}}{M_{\text{initial}}} \times 100 \qquad (3.1)$$

Where: $M_{extract}$ = Mass of peanut oil extracted from the sample (g)

$M_{initial}$ = Mass of peanut powder sample used (g)

## 3.4 Analysis of Chemical Constituents of Peanut Oil

### 3.4.1 GC-MS Analysis

GC–MS analyses of the peanut oil will be carried out on a Varian 240-MS GC–MS, outfitted with an auto injector and a capillary column. The flow rate for the helium carrier gasoline was 1.0 mL/min. The injection temperature used to be 250 °C. A 0.4 µL sample was injected in the split mode with a split ratio of 40:1. The temperature will be once programmed from 50 to 230 °C at a rate of 10 °C/min, then from 230 to 280 °C at a rate of 20 °C/min and subsequently held isothermal for 10 min. Ionization voltage (EI) used to be 70 eV. Ion source and interface temperature was 250 °C. Scan mass range was once 50–450 m/z. Solvent lengthen time used to be 1 min. Alkanes (C8-C40) have been used as references in the calculation of retention indices (RI). The aspects of peanut oils have been recognized through evaluation of their mass spectra with those of Finnigan Mainlib Library and Replib Library Databases (Thermo Fisher Scientific Inc., MA, USA), and further confirmed by contrast of their RI.

## 3.5 Kinetics Study of Peanut Oil Extraction

### 3.5.1 Mathematical Study

There are two mechanism that occur in the peanut oil extraction process. At the first moment of extraction oil which is in the washing step, it can be considered that the oil in the surface of the solid fibre will be removed by using the solvent. For the second part, the extraction of the oil that remains in the cells is carried out by using diffusion method. The hyperbolic model is the one of the kinetic model that used in extraction oil as pelegs model (Menkiti, Agu, & Udeigwe, 2015):

$$q = \frac{C_2 t}{1 + C_2 t} \tag{3.2}$$

The extraction is first – order at the beginning and decrease to zero – order in the later phase of the process. When $C_2$ t less than 1.

$$q \approx C_1 t \tag{3.3}$$

And when $t \longrightarrow \infty$, the equilibrium is reached ($q_i - q_e$). So,

$$q_e = \frac{q_e}{q_o} = \frac{C_1}{C_2} \tag{3.4}$$

Equation (3.5) is obtained when hyperbolic kinetic model equation is linearized.

$$\frac{1}{q} = \frac{1}{C_1} x \frac{1}{t} + \frac{C_2}{C_1} \quad (3.5)$$

The plot of 1/q that is 1/oil yield against 1/t gives slopes as $1/C_1$ and intercepts as $C_2/C_1$ whereas $C_1$ and $C_2$ are hyperbolic model parameters which are extraction rate at the beginning ($min^{-1}$) and constant related to maximum extraction yield ($min^{-1}$), respectively.

### 3.5.2 Statistical Analysis

The model performance had been evaluated through calculating the mean relative percentage deviation (MRPD) value as the following equation (Fuad & Karim, 2017):

$$\text{MRPD } (\%) = \frac{100}{N} \text{ x } \sum \frac{|\text{Y-Y}_p|}{Y} \quad (3.6)$$

Where N can be defined as the total number of experimental data. Y and $Y_p$ are yield of peanut oil for experimental and prediction, respectively. If the percent value of MRPD is less than 10%, the mathematical model are considered suitable to use in describing extraction oil process.

### 3.5.3 Calculation of Activation Energy

The relation between of rate of constant and extraction temperature can be expressed by the Arrhenius Equation as following:

$$k = A \exp\left[\frac{-E}{RT}\right] \quad (3.7)$$

Where:

k= The rate constant of extraction ($min^{-1}$)

A = The temperature independent factor ($min^{-1}$)

E = The activation energy (J $mol^{-1}$)

R = Gas constant (8.314 J $mol^{-1}$ $K^{-1}$)

T = Absolute temperature in K

# CHAPTER 4:

# RESULTS AND DISCUSSION

## 4.0 Introduction

The observed results were tabulated, analysed statistically, and discussed under this chapter. Generally, this chapter contain a few section which is optimisation of oil extraction from peanut, fatty acid analysis, kinetic of peanut oil extraction and calculation of activation energy.

## 4.1 Optimisation of peanut oil extraction

### 4.1.1 Effect of Solvent

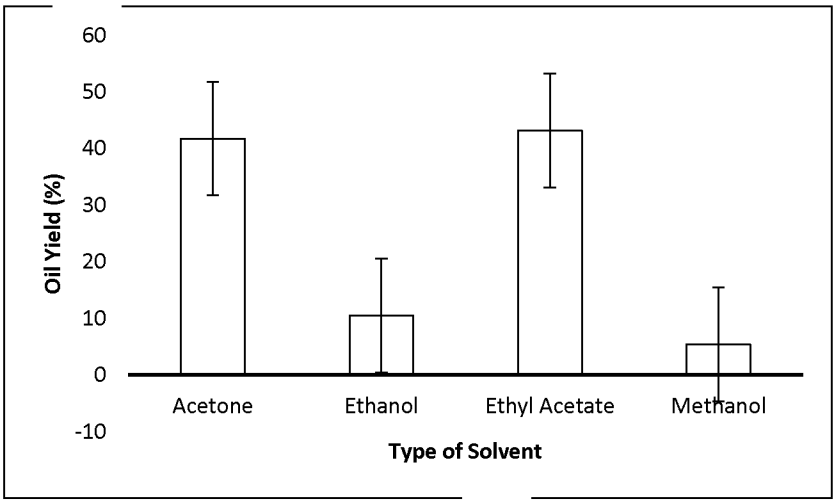


*Figure 5-1* Effect of solvent on oil yield using ultrasound technology with S/S of 10:1 for 30 min

The extraction yield with ethyl acetate was found to be the highest from others (43.2%). Then, it was followed by acetone (40.3%), ethanol (10.5%) and then methanol (5.4%) under similar conditions. According to (Tian et al., 2013) reported that the efficiency of ethyl acetate as solvent for the pomegranate seed oil extraction is higher. The polarity of the solvent is the one of the factor that influenced extraction yield (Silva, Garcia, & Zanette, 2016). Based on the polarity index (Sadek, 2002), ethyl acetate is slightly less polar than acetone, ethanol and methanol where the value of polarity indexes of ethyl acetate, acetone, ethanol and methanol are 4.4, 5.1, 5.2 and 5.1, respectively. So, it can be said that ethyl acetate extracted more oil than others.

According to (A. Abdolshahi, M.H. Majd, J.S. Rad, et al., 2015), ethyl acetate is the is known to be a good solvent for extracting unsaturated fatty acid. For the other studies from (Silva et al., 2016) reported that the performance of ethyl acetate as solvent for chia seed oil extraction gave the highest oil yield than isopropanol.

### 4.1.2 Effect of extraction Time

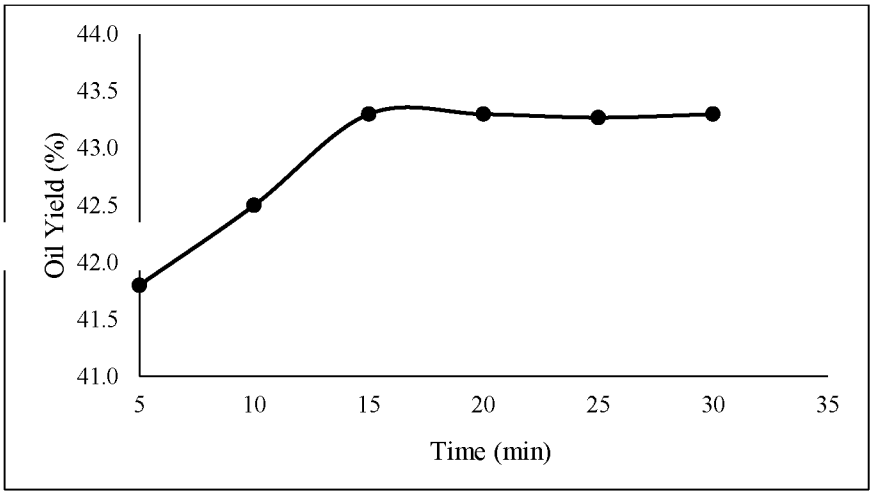


*Figure 5-2* The effect of reaction time on oil yield that using solvent of ethyl acetate with S/S ratio of 10:1 at temperature of 40°C that assisted by ultrasound technology.

The effect of extraction time on oil yield was shown in Figure 3. This figure show the comparison of extraction with ethyl acetate as solvent, S/S ratio of 10:1 for different treatment time from 5 to 30 min with steps size of 5 min. From the graph, oil yield increased significantly in the initial 15 min from 41.8% to 43.3%. After 15 min, oil yield reached an equilibrium and significantly decrease to 43.2% until 30 min. All peanut cell wall cracked completely within the first 15 min from the acoustic cavitation effect, leading to good penetration of the solvent into the cell (S. Hemwimol, P. Pavasant, 2006) and enhancing the transfer of dissolved oil out of the solid structure (M. Palma, 2002). As the time increased, the cell wall of peanut was ruptured that resulting to the lowering the solvent's permeability into cell structures. In addition, target components also re-adsorb into the ruptured tissue particles due to their relatively large specific surface areas, lowering oil yield (L. Paniwnyk, E. Beaufoy, J.P. Lorimer, 2001). A similar trend has also been suggested by (Liu et al., 2017) and (Perrier et al., 2017).

### 4.1.3 Effect of extraction temperature.

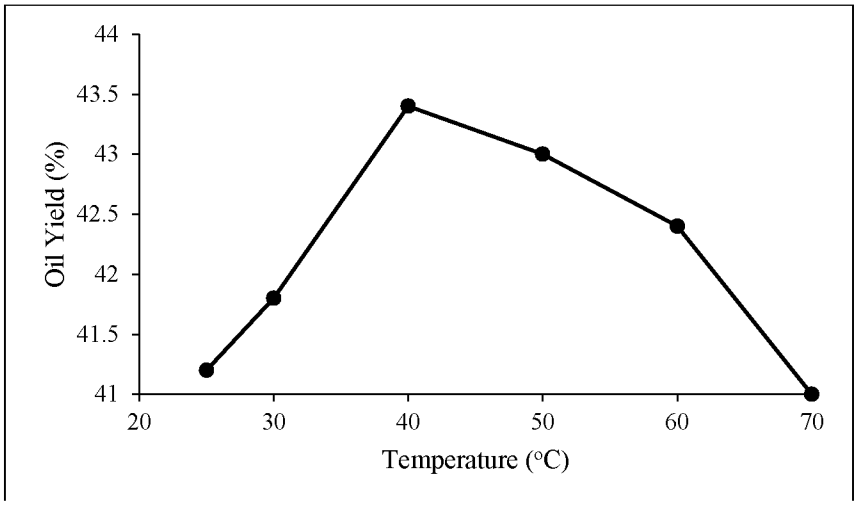


*Figure 5-3* The effect of reaction temperature on oil yield that using solvent of ethyl acetate with S/S ratio of 50mL/g for 15 min that assisted by ultrasound technology.

A range of temperature treatment which are from 25°C to 70°C were employed for 15 min assisted by ultrasonic sound with S/S ratio of 10:1 to assess their capabilities on the extraction oil from peanut. Result from the graph show the yield of oil increased rapidly as temperature increase from temperature of 25°C to 40°C (41.2% to 43.4%). However, when the temperature exceeded 40°C, the oil yield decrease significantly from 43.0% to 41.0%. As temperature increased, it will attribute to the increase solubility of peanut oil in solvent which is ethyl acetate. Moreover, when the temperature exceed the optimum temperature, the oil yield will become decrease significantly due to the increasing of mass transfer was resulted from the decreasing of viscosity and density of solvent (Z.S. Zhang, L.J. Wang, D. Li, et al., 2008) (Shalmashi, 2009). The number of cavitation bubbles within the fluid increased creating a cohesive force reducing the tensile strength of the liquid as a result of decreased viscosity of solvent (M. Palma, 2002) (M. Toma, M. Vinatoru, L. Paniwnyk, 2001). The same order of efficiency was also obtained by (Tian et al., 2013).

### 4.1.4 Effect of solvent to solid ratio

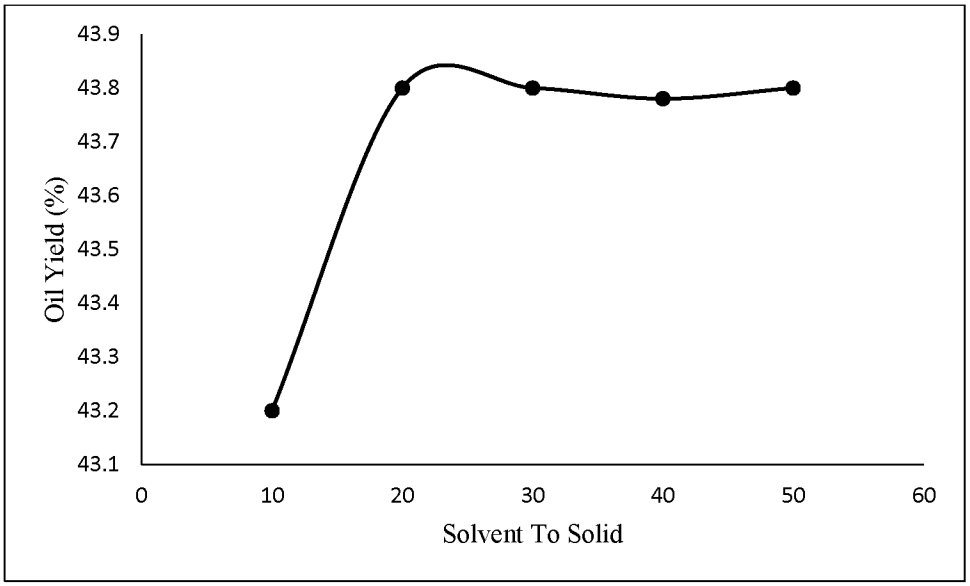


*Figure 5-4* Effect of solvent to solid ratio on the yield of oil at temperature of 40°C for 15 min

Five solvent to solid ratios of the effects of using ethyl acetate on oil yield were studied including 10:1, 20:1, 30:1, 40:1 and 50:1 at 40°C for 15 min, to determine the capabilities of the ratio of solvent to solid on oil extraction. Graph of Figure 4 shows the oil yield increase from 43.2 to 43.8% with the ratio of solvent to solid increasing from 10:1 to 20:1. However, when the solvent to solid ratio exceeded 20:1, the oil yield is not significantly increase, a larger amount of solvent will not change the driving force (S.S. Herodez, M. Hadolin, M. Skerget, 2003). With increasing of the solvent to solid ratio, the oil concentration between the extraction liquid and the materials also increase rapidly due to the effect of rate of diffusion that led to an enhancing of oil yield (Q.-A. Zhang, Z.-Q. Zhang, X.-F. Yue, et al., 2009). However, the excessive of solvent would give the effect of constant due to requirement of more energy and time to reflux the extraction solution. Hence, the optimum cycles for extraction could not be reached. Similar resulted had also been obtained by (Sayyar et al., 2009) and (Tian et al., 2013).

## 4.2 Kinetic Study of Oil Extraction

### 4.2.1 Effect of extraction temperature

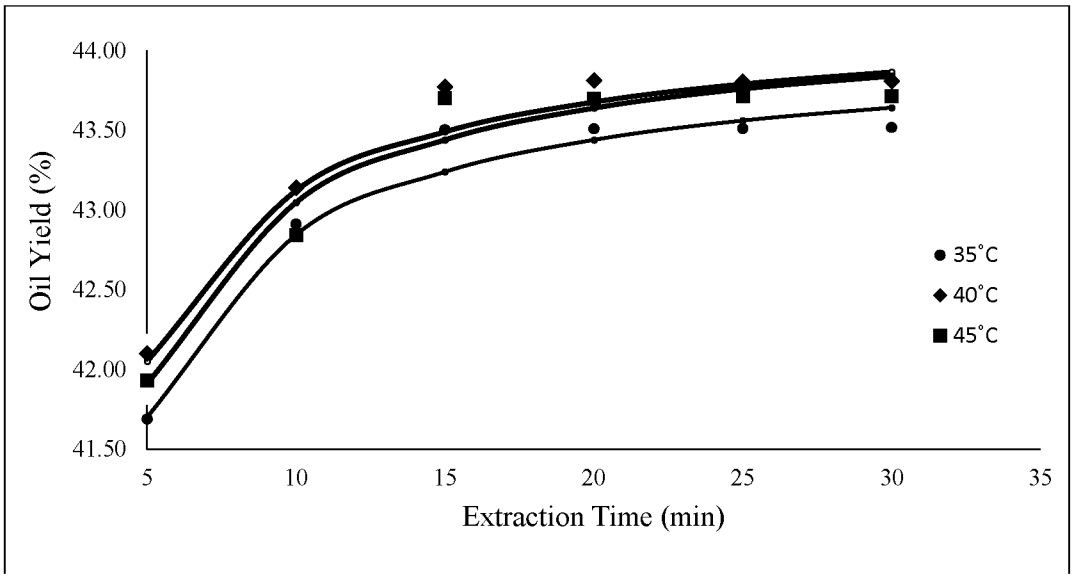


*Figure 5-5* Hyperbolic model kinetic of Peanut oil at different temperature using solvent of ethyl acetate, solvent to solid ratio of 20:1 for 30 min.

*Table 5-1*
Extraction rate and maximum extraction yield rate for different extraction temperature

| Extraction temperature (°C) | Extraction rate, $C_1$ ($min^{-1}$) | Maximum extraction yield rate, $C_2$ ($min^{-1}$) | MRPD | $R^2$ |
|---|---|---|---|---|
| 35 | 1.5625 | 0.03547 | 0.2243 | 0.9641 |
| 40 | 1.6949 | 0.03831 | 0.2022 | 0.9567 |
| 45 | 1.5873 | 0.03587 | 0.2759 | 0.9517 |

In the peanut oil extraction that assisted by ultrasound technology, it can be seen that the oil yield of peanut was increased rapidly at the beginning and then, reached equilibrium value with the length of extraction time. Hyperbolic kinetic model was applied to determine and study the kinetic model on the peanut oil extraction that assisted by ultrasound technology. To study the hyperbolic kinetic model on the extraction of the peanut oil, it can be done by plotting the graph between 1/q and 1/t. The slope and intercept from plotted graph were used to determine value of extraction rate, $C_1$ and maximum extraction yield rate, $C_2$. From the result obtained, extraction

rate, C1 were 1.5625, 1.6949 and 1.5873 $min^{-1}$ for extraction temperature of 35°C, 40°C and 45°C, respectively.

Basically, the increment of extraction rate $C_1$ and maximum extraction yield rate $C_2$ occurred with the increasing extraction temperature as shown in Table 1. However, when temperature exceed to 40°C, the value of extraction rate decreased significantly due to the massive amount of bubbles were formed (Parthiban & Perumalsamy, 2016). From that, they had collapsed with low of intensity. Moreover, when temperature is too high, it can caused the decreasing surface tension of the extracting solvent and automatically, it will affect the formation of bubbles and collapse (Nwabanne, 2012).

From the Table 1, it can be seen that hyperbolic kinetic model for peanut oil extraction had coefficient of determination ($R^2$) of 0.9883, 0.9826 and 0.9589 for extraction temperature of 35 °C, 40 °C, 45°C, respectively. While, the value of MRPD were 0.2243, 0.2022 and 0.2759 for temperature of 35°C, 40 °C and 45 °C, respectively. Hence, it can be said that hyperbolic kinetic model is able to present well the experimental results of peanut oil. This is also been supported by (Menkiti et al., 2015) which states that hyperbolic kinetic model reasonably described TC kernel oil extraction.

### 4.2.2 Effect of solvent to solid ratio

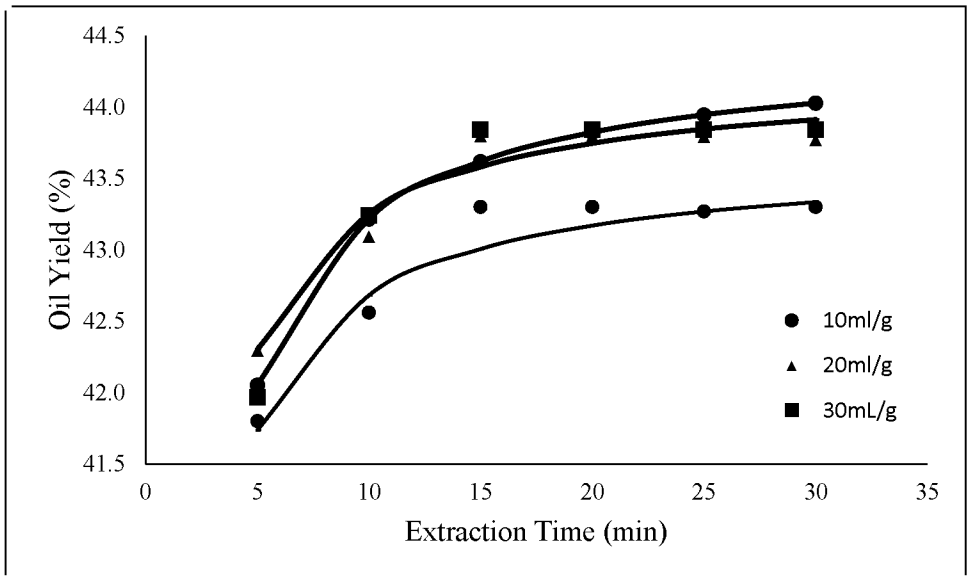


*Figure 5-6* Hyperbolic model kinetic of Peanut oil at different solvent to solid ratio using ethyl acetate as solvent with temperature of 40°C for 30 min.

*Table 5-2*

Extraction rate and maximum extraction yield rate for different solvent to solid ratio (mL/g)

| Solvent to solid ratio ( mL/g) | Extraction rate, $C_1$ ($min^{-1}$) | Maximum extraction yield rate, $C_2$ ($min^{-1}$) | MRPD | $R^2$ |
|---|---|---|---|---|
| 10 | 1.8868 | 0.04321 | 0.2500 | 0.9362 |
| 20 | 1.9231 | 0.04346 | 0.2337 | 0.9501 |
| 30 | 1.5625 | 0.03516 | 0.2467 | 0.9634 |

The effect of solvent to solid ratio (mL/g) was studied between 10:1 to 30:1 at constant of extraction temperature 40°C. From the result obtained in Figure 6 shows that the oil yield increased rapidly with solvent to solid ratio at the early beginning of the process from 1 min until 15 min and then the oil yield remain constant after 15 min of extraction time. The extraction rate of the hyperbolic kinetic model were determined from the data of experimental are shown in Table 2. From the result obtained, extraction rate, C1 were 1.8868, 1.9231 and 1.5625 $min^{-1}$ for solvent to solid ratio of 10mL/g, 20mL/g and 30mL/g, respectively.

An increasing solvent to solid ratio from 10:1 to 20:1 with increasing the extraction rate, $C_1$ and maximum extraction yield rate, $C_2$. However, when the solvent to solid ratio exceed 30:1, the extraction rate, $C_1$ and maximum extraction yield rate, $C_2$ decrease significantly. As the solvent to solid ratio increases, the gradient of concentration between solute and solvent is higher. Hence the mass transfer increases. Once there is a sufficient amount of solvent available for extraction, further addition of solvent does not affect the extraction yield (Charpe & Rathod, 2016). This is also supported by (Lin et al., 2012) which stated the excessive solvent would not increase the driving force effectiveness due to the limitation of the oil transfer is more confined to the solid interior.

The values of determination coefficient ($R^2$) for solvent to solid ratio of 10:1, 20:1 and 30:1 are 0.9767, 0.9515, and 0.9656, respectively. The higher value of the determination coefficient indicated that experimental data fit the model very well. While, the value of MRPD for solvent to solid ratio of 10mL/g, 20mL/g and 30mL/g are 0.2500, 02337 and 0.2467. It shows that the model is reasonably to be used in describing the extraction of peanut oil.

## 4.3 Calculation of activation energy.

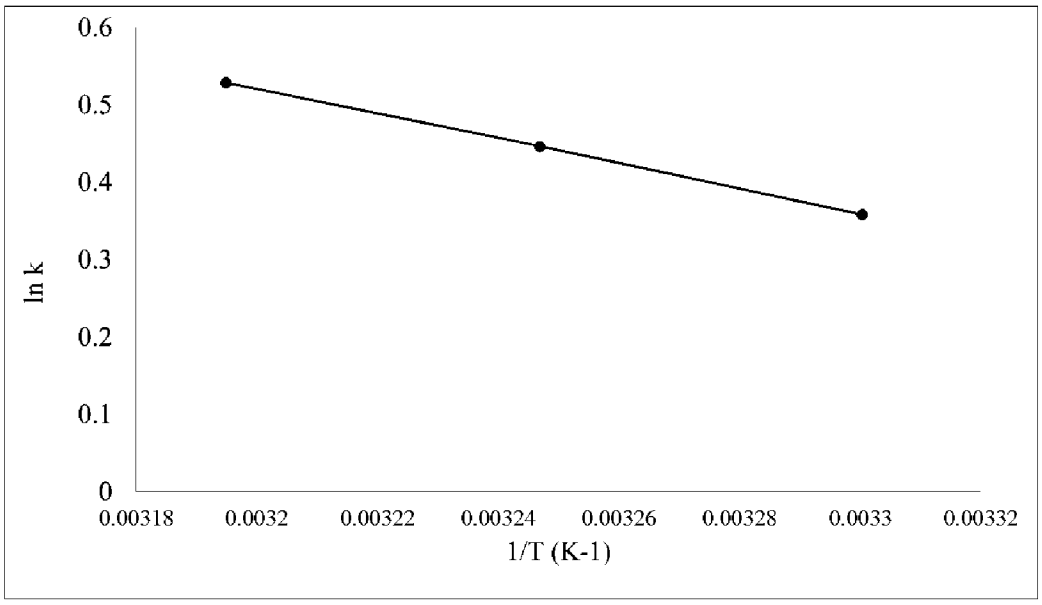


*Figure 5-7* Linear relationship between extraction rate constant of hyperbolic model, ln (k) and temperature for peanut oil extraction.

The extraction rate of hyperbolic kinetic model increase with the extraction temperature as shown in Table 1 and the changes can be described by the Arrhenius Equation. From the Figure 7, it shows a linear representation of Arrhenius Equation. The relationship between k and T can be defined by the linearized Arrhenius Equation (Eq. 3.3), where A and activation energy were found to be 292.5396 $min^{-1}$ and 13402.17 J $mol^{-1}$ respectively obtained from the intercept and the slope of the plotted graph.

## 4.4 Composition analysis and properties of peanut oil.

Fatty acid composition of peanut oil was analysed by GC-MS. From the analysis in Table 3, it shows that some of fatty acid constituents in the oil obtained such as lauric acid or known as dodecanoic acid. However, the result obtained did not show the real result like theoretical of fatty acid composition of peanut oil. It possibly due to the damaged, contaminated peanut oil sample or inefficiency of GC-MS that influenced the fatty acid composition of peanut oil. According to (Mzimbiri, Shi, Liu, & Wang, 2014), the authors had stated that the main composition of fatty acid is mono unsaturated fatty acid which oleic acid (40-50%). Then, it followed by linoleic acid and palmitic acid.

*Table 5-3*
Chemical constituents of peanut oil

| No | Retention Time (min) | Compound | Area (%) |
|---|---|---|---|
| 1 | 3.32 | 2,2'-Bifuran, octahydro | 0.62 |
| 2 | 3.33 | 2,2'-Bifuran, octahydro | 0.67 |
| 3 | 3.38 | Butane, 2,2,3-trimethyl | 13.1 |
| 4 | 3.40 | 3-methyl Pentane | 19.08 |
| 5 | 3.401 | N-Methoxydiacetamide | 12.8 |
| 6 | 3.418 | 1,3,5-Norcaratriene | 0.11 |
| 7 | 3.435 | 2-Propenoic acid, ethyl ester | 0.2 |
| 8 | 3.458 | Aziridine, 2-methyl- | 0.22 |
| 9 | 3.462 | 3-ethyl-2,2-dimethyl Pentane, | 0.32 |
| 10 | 3.464 | Propane, 1-(methylthio)- | 0.17 |
| 11 | 3.543 | methyl-Cyclopentane, | 8.59 |
| 12 | 3.548 | 2-Propynal | 1.06 |
| 13 | 3.739 | Cyclohexane | 0.14 |
| 14 | 31.361 | Dodecanoic acid | 0.38 |
| 15 | 31.371 | Dodecanoic acid, 1-methylethyl ester | 1.89 |

# CHAPTER FIVE:

# CONCLUSION AND RECOMMENDATIONS

## 5.1 Conclusion

There are four main type of operating parameter that influenced the peanut oil extraction that assisted by ultrasonic technology. The optimum condition for the solid liquid extraction from peanut in lab scale were obtained at temperature of 40°C, extraction time of 15 min, solvent to solid ration of 20:1 and ethyl acetate as the solvent used. Ethylene acetate was the most effective solvent for oil extraction followed by acetone, ethanol, and methanol. The hyperbolic kinetic model can represent well the experimental results of peanut oil extraction by ultrasound technology. The experimental data agrees well with the hyperbolic kinetic model with the determination coefficient in range of 0.95- 0.96.

## 5.2 Recommendation

For future work, the implementation ultrasound power as parameter need to be applied to determine the optimum ultrasound power for peanut oil extraction. UAE is seen as an ideal option for the edible oil industry because of improvements in efficiency and speed and because it can be performed at low operation temperatures which avoids thermal damage to the extracts and preserves the structural and molecular properties of bioactive compounds (Z.S. Zhang, L.J. Wang, D. Li, S.S. Jiao, et al., 2008) (Vilkhu et al., 2008) (Vinatoru, 2001). Besides that, surface area of solid should be studied as experimental parameters. From that, it can determine the optimum surface area of peanut which can optimize the oil extraction process. The size of solid can be varied in size of less than 0.5mm, between 0.5 -0.75 mm and bigger than 0.75mm.

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